

Search Strategy

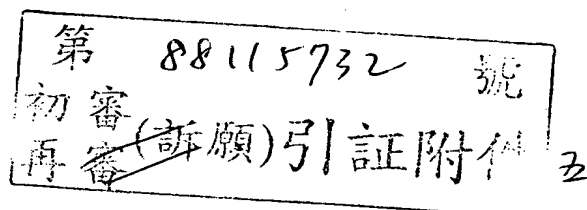
(Word)(tetrahydronaphthalene) and (Word)(liquid crystal)

127:364226

**Liquid crystal** composition having positive dielectric anisotropy and electrooptical display device using the **liquid crystal** composition.

Ichinose, Hideo; Takashima, Akio (Merck Patent GmbH, Germany). Brit. UK Pat. Appl. GB 2310669 A1 3 Sep 1997, 39 pp. (United Kingdom). CODEN: BAXXDU. CLASS: ICM: C09K019-30. ICA: C09K019-02. APPLICATION: GB 97-3021 13 Feb 1997. PRIORITY: EP 96-102880 27 Feb 1996. DOCUMENT TYPE: Patent CA Section: 74 (Radiation Chemistry, Photochemistry, and Photographic and Other Reprographic Processes) Section cross-reference(s): 75

There are provided both a liq. **crystal** compn. having a pos. dielec. anisotropy and an electrooptical display device comprising a realignment layer (for realigning liq. crystals whose field exhibits a significant component parallel to the liq. **crystal** layer) and the liq. **crystal** compn. The liq. **crystal** compn. contains at least one mesogenic compd. carrying a 3,4,5-trifluorophenyl group and/or at least one mesogenic compd. of the formula  $R^1(A^1Z^1)_m A^2 R^2$  wherein  $R^1$  and  $R^2$  are H or an alkyl or alkenyl radical of 1 to 15 carbon atoms which is unsubstituted or monosubstituted by CN or  $CF_3$  or at least monosubstituted by halogen atoms, it also being possible for one or more  $CH_2$  groups in these radicals to be replaced by O, S, 1,3-cyclobutanediyl, CO,  $CO_2$ , OCO, or  $OCO_2$  in such a manner that oxygen atoms are not linked directly to one another,  $A^1$  and  $A^2$  are (a) a trans-1,4-cyclohexylene radical or a 1,4-cyclohexenylene radical in which one or more nonadjacent  $CH_2$  groups can also be replaced by O and/or S, (b) a 1,4-phenylene radical in which one or two CH groups can also be replaced by N, or (c) a radical selected from the group consisting of 1,4-bicyclo[2.2.2]octylene, piperidine-1,4-diyl, naphthalene-2,6-diyl, decahydronaphthalene-2,6-diyl, and 1,2,3,4-tetrahydronaphthalene-2,6-diyl, where the radicals (a) and (b) can be substituted by one or two F atoms,  $Z^1$  is a single bond,  $CO_2$ , OCO,  $CH_2O$ ,  $OCH_2$ ,  $CH_2CH_2$ ,  $CH=CH$ ,  $C\equiv C$ ,  $(CH_2)_4$ , or  $CH=CHCH_2CH_2$ , and m is 1, 2, or 3.



# 5

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C4X X12  
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(56) Documents Cited

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GB 2234507 A	GB 2220658 A	GB 2218523 A
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(58) Field of Search

UK CL (Edition O ) C4X X12  
INT CL<sup>6</sup> C09K 19/00


(54) Liquid-crystalline medium having positive dielectric anisotropy and an electro-optical liquid-crystal display containing such a medium

(57) There is provided both a liquid-crystalline medium (as defined below) and an electro-optical liquid-crystal display comprising a realignment layer (for realigning liquid crystals whose field exhibits a significant component parallel to the liquid-crystal layer) and containing a liquid-crystalline medium having positive dielectric anisotropy, the medium containing at least one mesogenic compound carrying a 3,4,5-trifluorophenyl group and/or at least one mesogenic compound of the formula I



in which

R<sup>1</sup> and R<sup>2</sup> are each, independently of one another,

H, an alkyl or alkenyl radical of 1 to 15 carbon atoms which is unsubstituted or monosubstituted by CN or CF<sub>3</sub> or at least monosubstituted by halogen, it also being possible for one or more CH<sub>2</sub> groups in these radicals to be replaced, in each case independently of one another, by -O-, -S-, , -CO-, -CO-O-, -O-CO- or -O-CO-O- in such a manner that oxygen atoms are not linked directly to one another,

A<sup>1</sup> and A<sup>2</sup> are each, independently of one another,

- (a) a trans-1,4-cyclohexylene radical or a 1,4-cyclohexenylene radical in which one or more non-adjacent CH<sub>2</sub> groups can also be replaced by -O- and/or -S-,
- (b) a 1,4-phenylene radical in which one or two CH groups can also be replaced by N,
- (c) a radical from the group consisting of 1,4-bicyclo[2.2.2]octylene, piperidine-1,4-diyl, naphthalene-2,6-diyl, decahydronaphthalene-2,6-diyl and 1,2,3,4-tetrahydronaphthalene-2,6-diyl,

(57) continued overleaf

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(57) cont

where the radicals (a) and (b) can be substituted by one or two fluorines,

$Z^1$  is  $-\text{CO}-\text{O}-$ ,  $-\text{O}-\text{CO}-$ ,  $-\text{CH}_2\text{O}-$ ,  $-\text{OCH}_2-$ ,  $-\text{CH}_2\text{CH}_2-$ ,  $-\text{CH}=\text{CH}-$ ,  $-\text{C}\equiv\text{C}-$  or a single bond, furthermore also  $-(\text{CH}_2)_4-$  or  $-\text{CH}=\text{CH}-\text{CH}_2\text{CH}_2-$ , and

m is 1, 2 or 3.

## Electro-optical liquid-crystal display


The invention relates to an electro-optical liquid-crystal display comprising a realignment layer for  
 5 realigning liquid crystals whose field exhibits a significant component parallel to the liquid-crystal layer and containing a liquid-crystalline medium having positive dielectric anisotropy, the medium containing at least one mesogenic compound carrying a  
 10 3,4,5-trifluorophenyl group and/or at least one mesogenic compound of the formula I



15 in which

$R^1$  and  $R^2$  are each, independently of one another,

20 H, an alkyl or alkenyl radical of 1 to 15 carbon atoms which is unsubstituted or monosubstituted by CN or  $CF_3$  or at least monosubstituted by halogen, it also being possible for one or more  $CH_2$  groups in these radicals to be replaced, in each case  
 25 independently of one another, by -O-,

-S-, , -CO-, -CO-O-, -O-CO- or -O-CO-O-

in such a manner that oxygen atoms are not linked directly to one another,

30  $A^1$  and  $A^2$  are each, independently of one another,

(a) a trans-1,4-cyclohexylene radical or 1,4-cyclohexenylene radical in which one or more non-adjacent  $CH_2$  groups can also be replaced by -O- and/or -S-,  
 35

(b) a 1,4-phenylene radical in which one or two CH groups can also be replaced by N,

(c) a radical from the group consisting of  
1,4-bicyclo[2.2.2]octylene, piperidine-  
1,4-diyl, naphthalene-2,6-diyl, deca-  
hydronaphthalene-2,6-diyl and 1,2,3,4-  
5 tetrahydronaphthalene-2,6-diyl,

where the radicals (a) and (b) can be substituted  
by one or two fluorines,

10  $Z^1$  is  $-CO-O-$ ,  $-O-CO-$ ,  $-CH_2O-$ ,  $-OCH_2-$ ,  $-CH_2CH_2-$ ,  
 $-CH=CH-$ ,  $-C\equiv C-$  or a single bond, furthermore  
also  $-(CH_2)_4-$  or  $-CH=CH-CH_2CH_2-$ , and

m is 1, 2 or 3.

15 In customary liquid-crystal displays (TN, STN, OMI, AMD-  
TN), the electrical fields for effecting realignment are  
for the most part generated perpendicular to the liquid-  
crystal layer.

20 International Patent Application WO 91/10936 discloses a  
liquid-crystal display in which the electrical signals  
are generated in such a manner that the electrical  
fields possess a significant component parallel to the  
25 liquid-crystal layer (IPS, in-plane switching). The  
principles of operating such a display are described,  
for example, by R.A. Soref in Journal of Applied  
Physics, Vol. 45, No. 12, pp. 5466-5468 (1974).

30 EP 0,588,568 discloses various possibilities of  
addressing such a display.

These IPS displays can be operated with liquid-  
crystalline materials which either have a positive or a  
35 negative dielectric anisotropy ( $\Delta\epsilon \neq 0$ ). The existing  
materials exhibit high values of dielectric anisotropy,  
which are achieved in particular by using liquid  
crystals having terminal cyano groups. However, these

compounds have an unfavourable effect on the resistance and the voltage holding ratio of the IPS display element. Moreover, using high proportions of polar compounds results in unfavourable viscosity values and  
5 high birefringences, which is unfavourable for many applications. Accordingly, the object was to disclose liquid-crystalline materials which are suitable for achieving relatively high voltage holding ratios, low birefringence and viscosity values and short switching  
10 times in IPS displays.

Surprisingly, this object was achieved by using liquid-crystalline materials which contain at least one compound carrying a 3,4,5-trifluorophenyl group and/or  
15 contain at least one mesogenic compound of the formula I.

Compounds of this type are disclosed, for example, in  
20 EP 0,132,553 and DE 2,636,684.

However, in these disclosures, there is no mention that the voltage holding ratio and the switching times of IPS displays can be improved by means of these substances.


25 Accordingly, the invention provides an electro-optical liquid-crystal display comprising a realignment layer for realigning liquid crystals whose field has a significant component parallel to the liquid-crystal layer and containing a liquid-crystalline medium having  
30 positive dielectric anisotropy, the medium containing at least one mesogenic compound carrying a 3,4,5-trifluorophenyl group and/or at least one mesogenic compound of the formula I



in which

R<sup>1</sup> and R<sup>2</sup> are each, independently of one another,

5 H, an alkyl or alkenyl radical of 1 to 15 carbon atoms which is unsubstituted or monosubstituted by CN or CF<sub>3</sub> or at least monosubstituted by halogen, it also being possible for one or more CH<sub>2</sub> groups in these radicals to be replaced, in each case independently of one another, by -O-,

10 -S-, , -CO-, -CO-O-, -O-CO- or -O-CO-O- in such a manner that oxygen atoms are not linked directly to one another,

A<sup>1</sup> and A<sup>2</sup> are each, independently of one another,

15 (a) a trans-1,4-cyclohexylene radical or 1,4-cyclohexenylene radical in which one or more non-adjacent CH<sub>2</sub> groups can also be replaced by -O- and/or -S-,

20 (b) a 1,4-phenylene radical in which one or two CH groups can also be replaced by N,

25 (c) a radical from the group consisting of 1,4-bicyclo[2.2.2]octylene, piperidine-1,4-diyl, naphthalene-2,6-diyl, decahydronaphthalene-2,6-diyl and 1,2,3,4-tetrahydronaphthalene-2,6-diyl,

30 where the radicals (a) and (b) can be substituted by one or two fluorines,

Z<sup>1</sup> is -CO-O-, -O-CO-, -CH<sub>2</sub>O-, -OCH<sub>2</sub>-, -CH<sub>2</sub>CH<sub>2</sub>-, -CH=CH-, -C≡C- or a single bond, furthermore also -(CH<sub>2</sub>)<sub>4</sub>- or -CH=CH-CH<sub>2</sub>CH<sub>2</sub>-, and

35

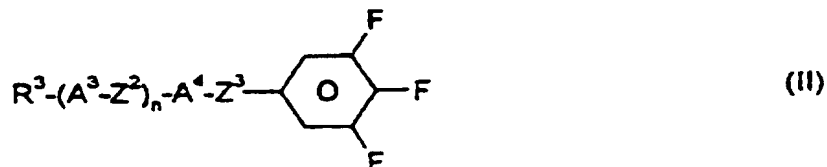
m is 1, 2 or 3.

Preferred embodiments are IPS displays where

a) the medium contains at least one compound of the formula I,

5

b) the medium contains at least one compound of the formula II



in which

10

$R^3$  has the meaning given for  $R^1$  and  $R^2$ ,

$A^3$  and  $A^4$  each have, independently of one another, the meaning given for  $A^1$  and  $A^2$ ,

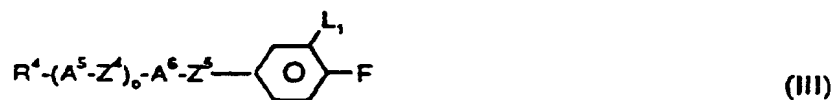
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$Z^2$  and  $Z^3$  each have, independently of one another, the meaning given for  $Z^1$ , and

$n$  is 0, 1 or 2;

20

c) the medium contains at least one compound of the formula III



in which

25

$R^4$  has the meaning given for  $R^1$ ,

$A^5$  and  $A^6$  each have, independently of one another, the meaning given for  $A^1$  and  $A^2$ ,

30

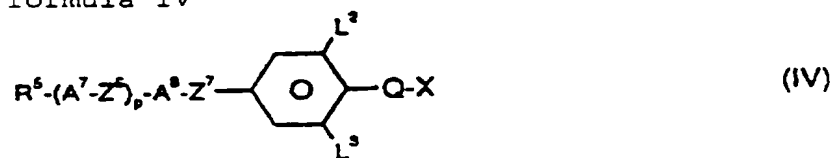
$Z^4$  and  $Z^5$  each have, independently of one another, the meaning given for  $Z^1$ ,



o is 0, 1 or 2, and

L<sup>1</sup> is H, F or Cl,

5 d) the medium contains at least one compound of the formula IV



in which

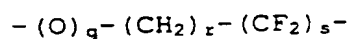
10 R<sup>5</sup> has the meaning given for R<sup>i</sup>,

A<sup>7</sup> and A<sup>8</sup> each have, independently of one another, the meaning given for A<sup>1</sup> and A<sup>2</sup>,

15 Z<sup>6</sup> and Z<sup>7</sup> each have, independently of one another, the meaning given for Z<sup>1</sup>,

L<sup>2</sup> and L<sup>3</sup> are each, independently of one another, H or F,

20 Q is a polyfluoroalkylene radical of the formula



25

in which

q is 0 or 1,

30 r is 0 or an integer between 1 and 6,

s is an integer between 1 and 6,

X is H, F or Cl, and

35

p is 0, 1 or 2,

- e) the medium contains at most 10% of liquid crystals carrying cyano groups.

5

Further preference is given to an IPS display where the pixels are addressed by an active matrix.

The invention also provides a liquid-crystalline medium having positive dielectric anisotropy which contains at least one compound carrying a 3,4,5-trifluorophenyl group, preferably a compound of the formula II, and at least one compound of the formula I, in particular one which contains

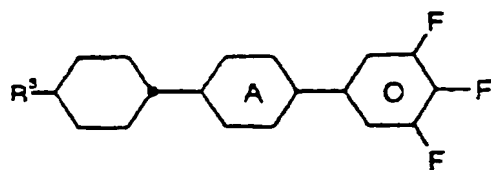
15

- 20 to 60, preferably 15 to 40, % by weight of at least one compound of the formula I,
- 10 to 60, preferably 20 to 50, % by weight of at least one compound of the formula II,
- 0 to 20, preferably 5 to 15, % by weight of at least one compound of the formula III and
- 25 - 0 to 40, preferably 20 to 30, % by weight of at least one compound of the formula IV.

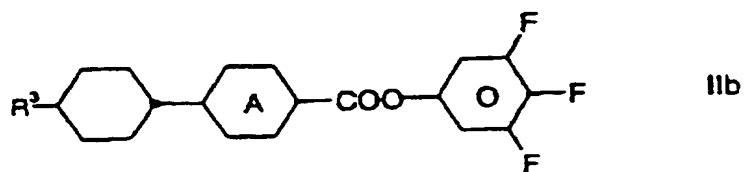
Preferably, the liquid-crystalline medium according to the invention contains:

30

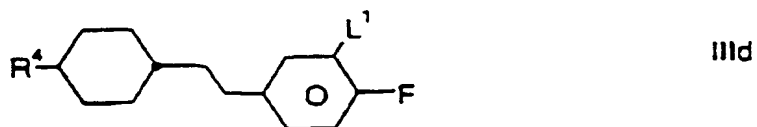
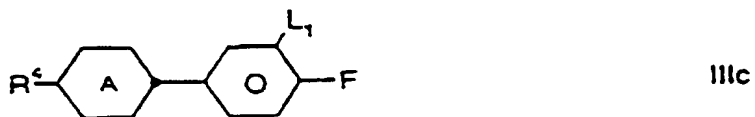
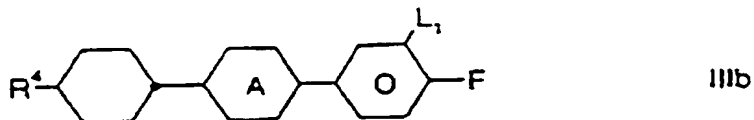
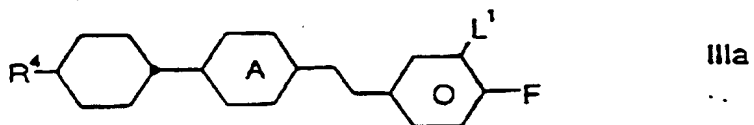
- at least one compound selected from formulae IIa and IIb



IIa

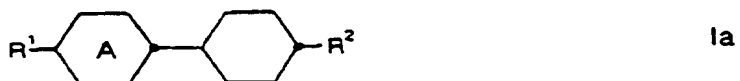


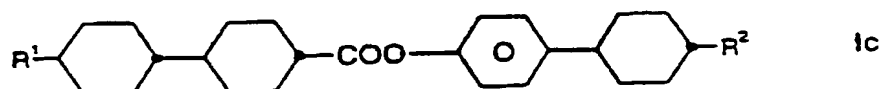
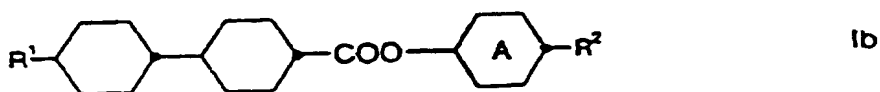
- at least one compound selected from formulae IIIa, IIIb, IIIc and IIId



5

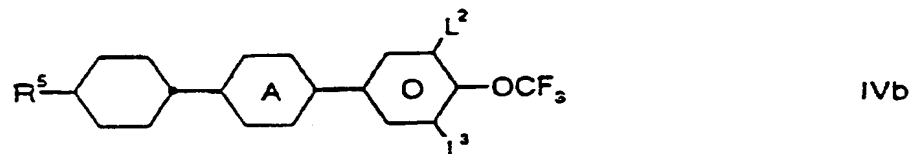
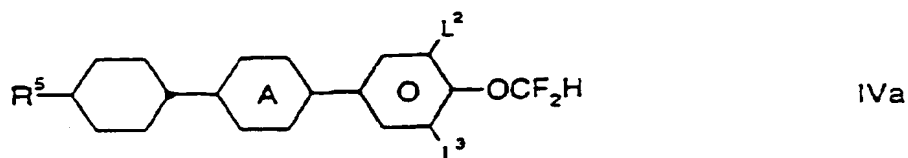
- at least one compound selected from formulae Ia, Ib and Ic



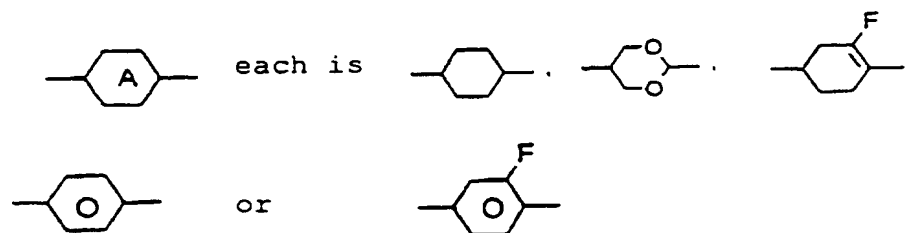


and, if desired,

- 5 - at least one compound selected from formulae IVa and IVb

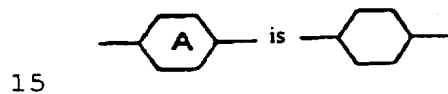


in which

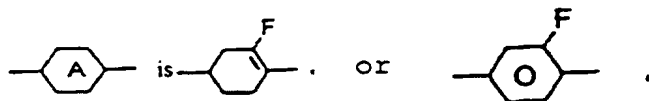


- 10 and R<sup>1</sup>, R<sup>2</sup>, R<sup>3</sup>, R<sup>4</sup>, R<sup>5</sup>, L<sup>1</sup>, L<sup>2</sup> und L<sup>3</sup> each have the meaning given.

In a particularly preferred embodiment, the media according to the invention contain at least one compound of the formula IIa in which



and at least one compound of the formula IIa in which

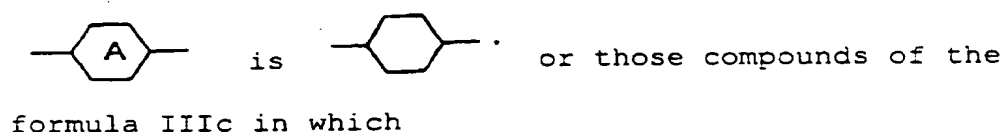


In a further preferred embodiment, the media according to the invention contain at least one compound of the formula IIa and at least one compound of the formula IIb.

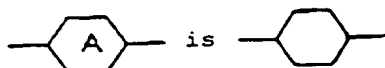
Further preference is given to media containing at least one compound of the formula IIIb in which

10

$L^1$  is F and

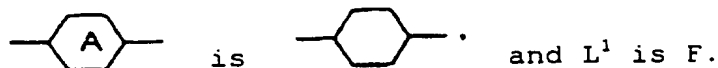


15  $L^1$  is H and



Particularly preferably, the media according to the invention contain a compound of the formula IIIa in which

20



Particular preference is given to media containing not more than 10%, in particular to those containing preferably 0 to 5% of liquid crystals carrying cyano groups.

25

Typically, the liquid-crystalline media according to the invention have a birefringence ( $\Delta n$ ) of  $< 0.12$ ,  $\Delta n$  is preferably between 0.06 and 0.1, in particular between 0.06 and 0.08.

5

Typically, the viscosity (at  $20^{\circ}\text{C}$ ) of the materials according to the invention is less than  $30 \text{ mm}^2 \text{ s}^{-1}$ , in particular between 10 and  $25 \text{ mm}^2 \text{ s}^{-1}$ . Typically, the specific resistivity of the materials according to the invention at  $20^{\circ}\text{C}$  is between  $5 \times 10^{10}$  and  $5 \times 10^{13} \Omega \cdot \text{cm}$ , particularly preferably the values are between  $5 \times 10^{11}$  and  $5 \times 10^{12} \Omega \cdot \text{cm}$ .

It has been found that a relatively small proportion of compounds of the formula II in a mixture with customary liquid-crystal materials, but in particular with one or more compounds of the formulae I, III and/or IV already leads to a significant increase in the holding ratio and to short switching times, while simultaneously giving rise to broad nematic phases having low smectic-nematic transition temperatures. In particular the use of compounds of the formula I enables the viscosity and the birefringence to be reduced, which is advantageous for certain applications. The compounds of the formulae I to IV are colourless, stable and readily miscible with one another and with other liquid-crystal materials.

The term "alkyl" covers straight-chain and branched alkyl groups having 1-7 carbon atoms, in particular the straight-chain groups methyl, ethyl, propyl, butyl, pentyl, hexyl and heptyl. Groups having 2-5 carbon atoms are usually preferred.

The term "alkenyl" covers straight-chain and branched alkenyl groups having 2-7 carbon atoms, in particular the straight-chain groups. Suitable alkenyl groups are  $\text{C}_2\text{-C}_7\text{-1E-alkenyl}$ ,  $\text{C}_4\text{-C}_7\text{-3E-alkenyl}$ ,  $\text{C}_5\text{-C}_7\text{-4-alkenyl}$ ,  $\text{C}_6\text{-C}_7\text{-5-alkenyl}$  and  $\text{C}_7\text{-6-alkenyl}$ , in particular  $\text{C}_2\text{-C}_7\text{-1E-}$

alkenyl, C<sub>4</sub>-C<sub>7</sub>-3E-alkenyl and C<sub>5</sub>-C<sub>7</sub>-4-alkenyl. Examples of preferred alkenyl groups are vinyl, 1E-propenyl, 1E-butenyl, 1E-pentenyl, 1E-hexenyl, 1E-heptenyl, 3-butenyl, 3E-pentenyl, 3E-hexenyl, 3E-heptenyl, 5 4-pentenyl, 4Z-hexenyl, 4E-hexenyl, 4Z-heptenyl, 5-hexenyl, 6-heptenyl and the like. Groups having up to 5 carbon atoms are in general preferred.

10 The term "fluoroalkyl" preferably covers straight-chain groups containing terminal fluorine, i.e. fluoromethyl, 2-fluoroethyl, 3-fluoropropyl, 4-fluorobutyl, 5-fluoropentyl, 6-fluorohexyl and 7-fluoroheptyl. However, other fluorine positions are not excluded.

15 The term "oxaalkyl" preferably covers straight-chain radicals of the formula C<sub>n</sub>H<sub>2n+1</sub>-O-(CH<sub>2</sub>)<sub>m</sub> in which n and m are each, independently of one another, 1 to 6. Preferably, n is 1 and m is 1 to 6.

20 Through a suitable choice of the meanings of R<sup>1</sup> and R<sup>5</sup>, it is possible to modify the response times, the threshold voltage, the steepness of the transmission characteristics etc. as desired. For example, 1E-alkenyl radicals, 3E-alkenyl radicals, 2E-alkenyloxy radicals  
25 and the like usually lead to shorter response times, improved nematic tendencies, and a higher ratio of the elastic constants k<sub>33</sub> (bend) and k<sub>11</sub> (splay) compared with alkyl or alkoxy radicals. 4-Alkenyl radicals, 3-alkenyl radicals and the like in general result in  
30 lower threshold voltages and smaller k<sub>33</sub>/k<sub>11</sub> values compared with alkyl and alkoxy radicals.

In general, a group -CH<sub>2</sub>CH<sub>2</sub>- in Z<sup>1</sup> or Z<sup>2</sup> leads to higher k<sub>33</sub>/k<sub>11</sub> values compared with a simple covalent bond.  
35 Higher k<sub>33</sub>/k<sub>11</sub> values make it possible, for example, to achieve flatter transmission characteristics in TN cells having a 90° twist (for obtaining grey hues) and steeper

transmission characteristics in STN, SBE and OMI cells (higher multiplex ratio) and vice versa.

5 The optimum mixing ratio of the compounds of the formulae I and II + III + IV depends largely on the desired properties, on the choice of components of the formulae I, II, III and/or IV and on the choice of any other components which may be present. Suitable mixing ratios within the abovementioned range can be easily  
10 determined from case to case.

The total amount of compounds of the formulae I to IV in the mixtures according to the invention is not critical. Accordingly, the mixtures can contain one or more  
15 further components for optimizing various properties. However, the observed effect on the holding ratio, the response times and the threshold voltage is generally the greater, the higher the total concentration of the compounds of the formulae II and III. The observed  
20 effect on the viscosity and the birefringence is generally the greater, the higher the total concentration of the compounds of the formula I.

In a particularly preferred embodiment, the media  
25 according to the invention contain compounds of the formula IV (preferably I and/or III) in which Q-X is  $\text{OCF}_3$  or  $\text{OCHF}_2$ . A favourable synergistic effect with the compounds of the formula II leads to particularly advantageous properties.

30 Preferably, the liquid-crystalline media according to the invention contain, in addition to one or more compounds of the formulae I, II, III and IV, 2 to 40, in particular 4 to 30 components, as further constituents.  
35 Most preferably, these media contain, in addition to one or more compounds according to the invention, 7 to 25 components. These further components are preferably selected from nematic or nematogenic (monotropic or



isotropic) substances, in particular from substances from the classes of azoxybenzenes, benzylideneanilines, biphenyls, terphenyls, phenyl or cyclohexyl benzoates, phenyl or cyclohexyl cyclohexanecarboxylates, phenyl or  
 5 cyclohexyl cyclohexylbenzoates, phenyl or cyclohexyl cyclohexylcyclohexanecarboxylates, cyclohexylphenyl benzoates, cyclohexylphenyl cyclohexanecarboxylates or cyclohexyl-phenyl cyclohexylcyclohexanecarboxylates, phenylcyclohexanes, cyclohexylbiphenyls, phenylcyclohexylcyclohexanes, cyclohexylcyclohexanes, cyclohexylcyclohexyl-  
 10 cyclohexenes, 1,4-bis(cyclohexyl)benzenes, 4,4'-bis(cyclohexyl)biphenyls, phenyl- or cyclohexylpyrimidines, phenyl- or cyclohexylpyridines, phenyl- or cyclohexyldioxanes, phenyl- or cyclohexyl-1,3-dithianes,  
 15 1,2-diphenylethanes, 1,2-dicyclohexylethanes, 1-phenyl-2-cyclohexylethanes, 1-cyclohexyl-2-(4-phenylcyclohexyl)ethanes, 1-cyclohexyl-2-biphenylethanes, 1-phenyl-2-cyclohexylphenylethanes, optionally halogenated stilbenes, benzyl phenyl ethers, tolans and substituted  
 20 cinnamic acids. The 1,4-phenylene groups in these compounds may also be fluorinated.

The most important compounds suitable as further constituents of media according to the invention can be  
 25 characterized by the formulae 1, 2, 3, 4 and 5:

$R'-L-E-R''$	1
$R'-L-COO-E-R''$	2
$R'-L-OOC-E-R''$	3
$R'-L-CH_2CH_2-E-R''$	4
$R'-L-C\equiv C-E-R''$	5

In the formulae 1, 2, 3, 4 and 5, L and E, which may be identical or different, are in each case, independently  
 30 of one another, a bivalent radical from the group formed by -Phe-, -Cyc-, -Phe-Phe-, -Phe-Cyc-, -Cyc-Cyc-, -Pyr-, -Dio-, -G-Phe-, -G-Cyc- and their mirror images, where Phe is unsubstituted or fluorine-substituted 1,4-

phenylene, Cyc is trans-1,4-cyclohexylene or  
1,4-cyclohexenylene, Pyr is pyrimidine-2,5-diyl or  
pyridine-2,5-diyl, Dio is 1,3-dioxane-2,5-diyl and G is  
2-(trans-1,4-cyclohexyl)ethyl, pyrimidine-2,5-  
5 diyl, pyridine-2,5-diyl or 1,3-dioxane-2,5-diyl.

One of the radicals L and E is preferably Cyc, Phe or  
Pyr. E is preferably Cyc, Phe or Phe-Cyc. The media  
according to the invention preferably contain one or  
10 more components selected from the compounds of the  
formulae 1, 2, 3, 4 and 5 in which L and E are selected  
from the group consisting of Cyc, Phe and Pyr and  
simultaneously one or more components selected from the  
compounds of the formulae 1, 2, 3, 4 and 5 in which one  
15 of the radicals L and E is selected from the group  
consisting of Cyc, Phe and Pyr and the other radical is  
selected from the group consisting of -Phe-Phe-, -Phe-  
Cyc-, -Cyc-Cyc-, -G-Phe- and -G-Cyc- and optionally one  
or more components selected from the compounds of the  
20 formulae 1, 2, 3, 4 and 5 in which the radicals L and E  
are selected from the group consisting of -Phe-Cyc-,  
-Cyc-Cyc-, -G-Phe- and -G-Cyc-.

In a smaller subgroup of the compounds of the formulae  
25 1, 2, 3, 4 and 5, R' and R" are in each case,  
independently of one another, alkyl, alkenyl, alkoxy,  
alkoxyalkyl, alkenyloxy or alkanoyloxy having up to  
8 carbon atoms. This smaller subgroup will be referred  
to in the following as group A and the compounds will be  
30 designated by the partial formulae 1a, 2a, 3a, 4a and  
5a. In most of these compounds, R' and R" are different  
from one another, one of these radicals usually being  
alkyl, alkenyl, alkoxy or alkoxyalkyl.

35 In another smaller subgroup of the compounds of the  
formulae 1, 2, 3, 4 and 5, referred to as group B, R" is  
-F, -Cl, -NCS or  $-(O)_iCH_3-(k+1)F_kCl_1$  where i is 0 or 1 and  
k+1 is 1, 2 or 3. The compounds in which R" has this

meaning are designated by the partial formulae 1b, 2b, 3b, 4b and 5b. Particular preference is given to those compounds of the partial formulae 1b, 2b, 3b, 4b and 5b in which R" is -F, -Cl, -NCS, -CF<sub>3</sub>, -OCHF<sub>2</sub> or -OCF<sub>3</sub>.

5

In the compounds of the partial formulae 1b, 2b, 3b, 4b and 5b, R' has the meaning given for the compounds of the partial formulae 1a-5a and is preferably alkyl, alkenyl, alkoxy or alkoxyalkyl.

10

In a further smaller subgroup of the compounds of the formulae 1, 2, 3, 4 and 5, R" is -CN. This subgroup will be referred to in the following as group C and the compounds of this subgroup will accordingly be described by the partial formulae 1c, 2c, 3c, 4c and 5c. In the compounds of the partial formulae 1c, 2c, 3c, 4c and 5c, R' has the meaning given for the compounds of the partial formulae 1a-5a and is preferably alkyl, alkoxy or alkenyl.

15

20

Apart from the preferred compounds from groups A, B and C, other compounds of the formulae 1, 2, 3, 4 and 5 having other variants of the intended substituents are also common. All these substances are available by methods known from the literature or in analogy thereto.

25

The media according to the invention preferably contain, in addition to compounds according to the invention of the formula I, one or more compounds selected from group A and/or group B and/or group C. The weight proportions of the compounds from these groups in the media according to the invention are preferably as follows:

30

group A:	0 to 90%, preferably 20 to 90%, in particular 30 to 90%
group B:	0 to 80%, preferably 10 to 80%,

35

group C:

in particular 10 to 65%  
0 to 10%, preferably 0  
to 5%,  
in particular 0 to 2%,

5

the sum of the weight proportions of the compounds from groups A and/or B and/or C present in the corresponding media according to the invention being preferably 5% to 90% and, in particular, 10% to 90%.

10

The media according to the invention preferably contain 1 to 40%, particularly preferably 5 to 30%, of compounds according to the invention. Further preference is given to media containing more than 40%, in particular 45 to 90%, of compounds according to the invention. The media preferably contain three, four or five compounds according to the invention.

15

The construction of the IPS display according to the invention corresponds to the conventional design for displays of this type, such as described in WO 91/10936 or EP 0,588,568. The term conventional design is here drawn widely and also covers all derivatives and modifications of the IPS display, in particular also, for example, matrix display elements based on poly-Si TFT or MIM.

20

25

However, an essential difference between the displays according to the invention and those which have been customary up to now is the choice of liquid-crystal parameters in the liquid-crystal layer.

30

The liquid-crystal mixtures which can be used according to the invention are prepared in a manner customary per se. As a rule, the desired amount of the components used in the lesser amount is dissolved in the components making up the principal constituent, advantageously at elevated temperature. It is also possible to mix

35

solutions of the components in an organic solvent, for example acetone, chloroform or methanol, and, after thorough mixing, to remove the solvent again, for example by distillation.

5

The dielectrics may also contain further additives known to those skilled in the art and described in the literature. For example, 0-15% of pleochroic dyes or chiral dopants can be added.

10

C denotes a crystalline phase, S a smectic phase,  $S_B$  a smectic B phase, N a nematic phase and I the isotropic phase.

15

$V_{10}$  denotes the voltage for 10% transmission (viewing direction perpendicular to the plate surface).  $t_{on}$  denotes the switch-on time and  $t_{off}$  the switch-off time at an operating voltage corresponding to 2.5 times the value of  $V_{10}$ .  $\Delta n$  denotes the optical anisotropy and  $n_o$

20

the refractive index (both at 589 nm).  $\Delta\epsilon$  denotes the dielectric anisotropy ( $\Delta\epsilon = \epsilon_{||} - \epsilon_{\perp}$  where  $\epsilon_{||}$  is the dielectric constant parallel to the longitudinal molecular axes and  $\epsilon_{\perp}$  is the dielectric constant perpendicular thereto. The electro-optical data were

25

measured in an IPS cell at 20°C unless expressly stated otherwise. The optical data were measured at 20°C unless expressly stated otherwise.

30 The IPS test cell used was one in accordance with WO 91/10936 of comb structure in which the electrodes and the electrode interspaces have a spacing of 20  $\mu\text{m}$ .

The layer thickness  $d$  of the liquid-crystal material is 5  $\mu\text{m}$ . The cell furthermore comprises:

35

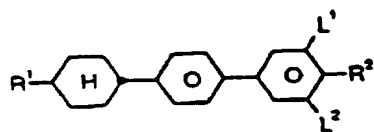
Starting twisting angle:	0°
Alignment angle:	5°
Surface tilt angle:	3°

In the "switched-off" state, the cells are dark.

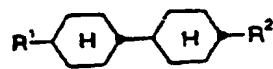
In the present invention and in the examples which follow, the structures of the liquid-crystal compounds  
5 are indicated by acronyms which can be transformed into chemical formulae with the aid of Tables A and B below. All radicals  $C_nH_{2n+1}$  and  $C_mH_{2m+1}$  are straight-chain alkyl radicals having n or m carbon atoms. The coding in Table B is self-explanatory. In Table A, only the acronym for  
10 the base structure is given. In individual cases, the acronym for the base structure is followed, separated by a hyphen, by a code for the substituents  $R^1$ ,  $R^2$ ,  $L^1$  and  $L^2$ :

Code for R <sup>1</sup> , R <sup>2</sup> , L <sup>1</sup> , L <sup>2</sup>	R <sup>1</sup>	R <sup>2</sup>	L <sup>1</sup>	L <sup>2</sup>
nm	C <sub>n</sub> H <sub>2n+1</sub>	C <sub>m</sub> H <sub>2m+1</sub>	H	H
nOm	C <sub>n</sub> H <sub>2n+1</sub>	OC <sub>m</sub> H <sub>2m+1</sub>	H	H
nO.m	OC <sub>n</sub> H <sub>2n+1</sub>	C <sub>m</sub> H <sub>2m+1</sub>	H	H
n	C <sub>n</sub> H <sub>2n+1</sub>	CN	H	H
nN.F	C <sub>n</sub> H <sub>2n+1</sub>	CN	F	H
nF	C <sub>n</sub> H <sub>2n+1</sub>	F	H	H
nOF	OC <sub>n</sub> H <sub>2n+1</sub>	F	H	H
nCl	C <sub>n</sub> H <sub>2n+1</sub>	Cl	H	H
nF.F	C <sub>n</sub> H <sub>2n+1</sub>	F	F	H
nN.F.F	C <sub>n</sub> H <sub>2n+1</sub>	CN	F	F
nCF <sub>3</sub>	C <sub>n</sub> H <sub>2n+1</sub>	CF <sub>3</sub>	H	H
nOCF <sub>3</sub>	C <sub>n</sub> H <sub>2n+1</sub>	OCF <sub>3</sub>	H	H
nOCF <sub>2</sub>	C <sub>n</sub> H <sub>2n+1</sub>	OCHF <sub>2</sub>	H	H
nS	C <sub>n</sub> H <sub>2n+1</sub>	NCS	H	H
rVsN	C <sub>7</sub> H <sub>23+1</sub> -CH=CH-C <sub>5</sub> H <sub>25</sub> -	CN	H	H
rEsN	C <sub>7</sub> H <sub>23+1</sub> -O-C <sub>2</sub> H <sub>25</sub> -	CN	H	H
nAm	C <sub>n</sub> H <sub>2n+1</sub>	COOC <sub>m</sub> H <sub>2m+1</sub>	H	H
nF.F.F	C <sub>n</sub> H <sub>2n+1</sub>	F	F	F
nCl.F.F	C <sub>n</sub> H <sub>2n+1</sub>	Cl	F	F
nCF <sub>3</sub> .F.F	C <sub>n</sub> H <sub>2n+1</sub>	CF <sub>3</sub>	F	F
nOCF <sub>3</sub> .F.F	C <sub>n</sub> H <sub>2n+1</sub>	OCF <sub>3</sub>	F	F
nOCF <sub>2</sub> .F.F	C <sub>n</sub> H <sub>2n+1</sub>	OCHF <sub>2</sub>	F	F
nOCF <sub>3</sub> .F	C <sub>n</sub> H <sub>2n+1</sub>	OCF <sub>3</sub>	F	H

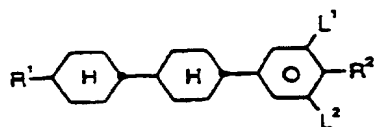
Table A:



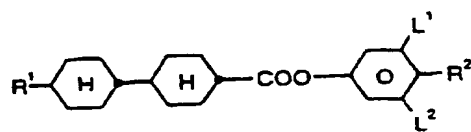
**BCH**



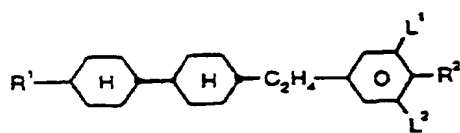
**CCH**



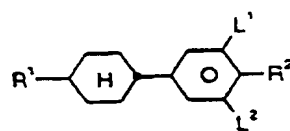
**CCP**



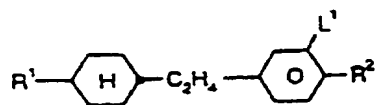
**CP**



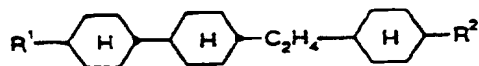
**ECCP**



**PCH**



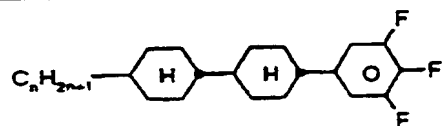
**EPCH**



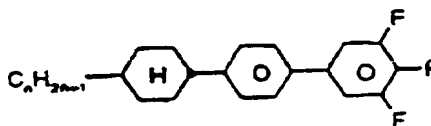
**CH**



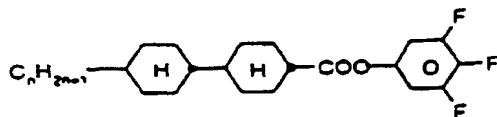
Table B:



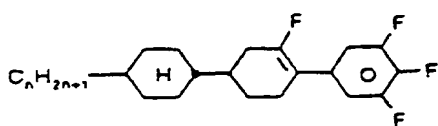
**CCP-nF.F.F**



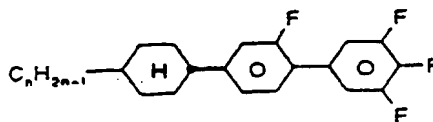
**BCH-nF.F.F**



**CCZU-n-F**



**CFU-n-F**



**CGU-n-F**

5 Even without any further explanations, it is assumed that a person skilled in the art can utilize the above description to the fullest extent. The preferred embodiments are therefore merely descriptive and should not be construed as limiting the disclosure in any way.

The complete disclosures of all applications, patent specifications and publications listed above and below and the corresponding applications

P 19,503,507 of 03.02.1995,  
P 19,509,791 of 17.03.1995,  
P 19,528,104 of 01.08.1995,  
P 19,528,106 of 01.08.1995,  
P 19,528,107 of 01.08.1995 and  
P 19,537,802 of 11.10.1995

are incorporated in this application by reference.

Example 1

15

An IPS display contains a nematic mixture exhibiting

	clearing point	+71°C
	$\Delta n$	0.0759
20	$n_0$	1.4793
	viscosity (20°C)	16 mm <sup>2</sup> s <sup>-1</sup>

and consisting of

25	PCH-7F	9.00
	PCH-302	4.00
	CCH-35	5.00
	CCH-303	6.00
	CCH-501	3.00
30	CCP-2OCF <sub>3</sub>	6.00
	CCP-3OCF <sub>3</sub>	6.00
	CCP-4OCF <sub>3</sub>	6.00
	CCP-5OCF <sub>3</sub>	6.00
	ECCP-3F.F	5.00
35	ECCP-5F.F	5.00
	CGU-2.F	4.00
	CGU-3.F	4.00
	CGU-5.F	4.00
	CCP-2F.F.F	10.00
	CCP-3F.F.F	12.00
	CCP-5F.F.F.	5.00

5 and shows high contrast and short switching times.

Example 2

An IPS display contains a nematic mixture exhibiting

5	clearing point	+80 °C
	$\Delta n$ [589 nm; 20 °C]	0.0761
	$n_o$	1.4769
	$\Delta \epsilon$ [1 kHz; 20 °C]	+5.0
	viscosity (20 °C)	16 mm <sup>2</sup> ·s <sup>-1</sup>

10

and consisting of

	PCH-7F	8.00 %
	PCH-302	5.00 %
15	CCH-301	12.00 %
	CCH-35	5.00 %
	CCP-20CF <sub>3</sub>	7.00 %
	CCP-30CF <sub>3</sub>	8.00 %
	CCP-40CF <sub>3</sub>	7.00 %
20	CCP-50CF <sub>3</sub>	8.00 %
	BCH-2F.F	3.00 %
	BCH-3F.F	4.00 %
	ECCP-3F.F	7.00 %
	ECCP-5F.F	8.00 %
25	CCP-2F.F.F	6.00 %
	CCP-3F.F.F	6.00 %
	CCP-5F.F.F	6.00 %

and shows high contrast and short switching times.

30

35

Example 3

An IPS display contains a nematic mixture exhibiting

5	clearing point	+71 °C
	$\Delta n$ [589 nm; 20 °C]	0.0843
	$n_o$	1.4861
	$\Delta \epsilon$ [1 kHz; 20 °C]	+6.3
	viscosity (20 °C)	16 mm <sup>2</sup> ·s <sup>-1</sup>

10

and consisting of

	PCH-7F	8.00 %
	PCH-301	7.00 %
15	PCH-302	6.00 %
	CCH-35	5.00 %
	CCP-20CF <sub>3</sub>	6.00 %
	CCP-30CF <sub>3</sub>	6.00 %
	CCP-40CF <sub>3</sub>	6.00 %
20	CCP-50CF <sub>3</sub>	6.00 %
	ECCP-3F	6.00 %
	CGU-2-F	6.00 %
	CGU-3-F	3.00 %
	BCH-3F.F	5.00 %
25	BCH-5F.F	4.00 %
	CCP-2F.F.F	10.00 %
	CCP-3F.F.F	11.00 %
	CCP-5F.F.F	5.00 %

30 and shows high contrast and short switching times.

35

Example 4

An IPS display contains a nematic mixture exhibiting

5	clearing point	+70 °C
	$\Delta n$ [589 nm; 20 °C]	0.00846
	$n_o$	1.4855
	$\Delta \epsilon$ [1 kHz; 20 °C]	+6.5
	viscosity (20 °C)	16 mm <sup>2</sup> ·s <sup>-1</sup>

10

and consisting of

	PCH-7F	8.00 %
	PCH-301	7.00 %
15	PCH-302	7.00 %
	CCH-35	3.00 %
	CCP-20CF <sub>3</sub>	6.00 %
	CCP-30CF <sub>3</sub>	6.00 %
	CCP-40CF <sub>3</sub>	6.00 %
20	CCP-50CF <sub>3</sub>	6.00 %
	ECCP-3F.F	6.00 %
	ECCP-5F.F	3.00 %
	CGU-2-F	5.00 %
	CGU-3-F	4.00 %
25	BCH-3F.F	4.00 %
	BCH-5F.F	4.00 %
	CCP-2F.F.F	9.00 %
	CCP-3F.F.F	11.00 %
30	CCP-5F.F.F	5.00 %

and shows high contrast and short switching times.

35

Example 5

An IPS display contains a nematic mixture exhibiting

5	clearing point	+96 °C
	$\Delta n$ [589 nm; 20 °C]	0.0859
	$n_o$	1.4795
	$\Delta \epsilon$ [1 kHz; 20 °C]	+4.1
	viscosity (20 °C)	16 mm <sup>2</sup> ·s <sup>-1</sup>

10

and consisting of

	CCH-301	9.50 %
	PCH-7F	7.00 %
15	CCP-20CF <sub>3</sub>	7.00 %
	CCP-30CF <sub>3</sub>	8.00 %
	CCP-40CF <sub>3</sub>	5.00 %
	CCP-50CF <sub>3</sub>	5.00 %
	CCP-3F.F.F	8.00 %
20	CCP-5F.F.F	4.00 %
	ECCP-3F.F	7.00 %
	PCH-302	10.00 %
	BCH-3F.F	7.00 %
	ECCP-3F	10.00 %
25	CBC-33F	4.00 %
	CBC-53F	3.00 %
	CCH-35	5.50 %

30

and shows high contrast and short switching times.

35

Comparative Example

10 An IPS display contains a nematic mixture exhibiting

clearing point	64°C
$\Delta n$	0.0813
$\Delta \epsilon$	+12.0

15 and consisting of

	ME2N.F	4.00
	ME3N.F	4.00
	ME5N.F	10.00
20	ME7N.F	6.00
	CCH-2	12.00
	CCH-3	12.00
	CCH-4	12.00
	CCP-3OCF <sub>3</sub>	6.00
25	CCP-5OCF <sub>3</sub>	6.00
	CH-33	3.00
	CH-35	3.00
	CCH-34	10.00
	CCH-35	4.00
30	CCH-303	4.00
	CCH-501	4.00

and shows a lower contrast, a higher holding ratio, a  
higher birefringence and a higher switching time than  
35 the display from Example 1.

Patent claims

1. Electro-optical liquid-crystal display


- 5           - comprising a realignment layer for realigning liquid crystals whose field exhibits a significant component parallel to the liquid-crystal layer and
- 10           - containing a liquid-crystalline medium having positive dielectric anisotropy, characterized in that the medium contains
- 15               (a)     at least one mesogenic compound carrying a 3,4,5-trifluorophenyl group
- and/or
- 20               (b)     at least one mesogenic compound of the formula I



in which

25            $R^1$  and  $R^2$  are each, independently of one another,

30           H, an alkyl or alkenyl radical of 1 to 15 carbon atoms which is unsubstituted or monosubstituted by CN or  $CF_3$  or at least monosubstituted by halogen, it also being possible for one or more  $CH_2$  groups in these radicals to be replaced, in each case independently of one another, by -O-,

-S-, , -CO-, -CO-O-, -O-CO- or -O-CO-O-

35           in such a manner that oxygen atoms are not linked directly to one another,



A<sup>1</sup> and A<sup>2</sup> are each, independently of one another,

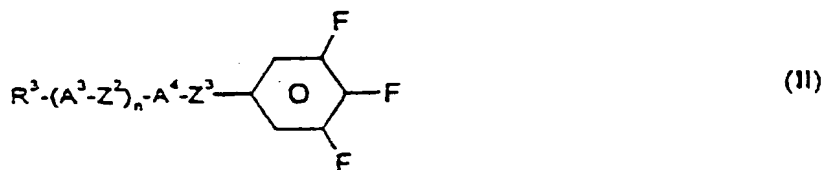
- 5 (a) a trans-1,4-cyclohexylene radical or a 1,4-cyclohexenylene radical in which one or more non-adjacent CH<sub>2</sub> groups can also be replaced by -O- and/or -S-,
- 10 (b) a 1,4-phenylene radical in which one or two CH groups can also be replaced by N,
- 15 (c) a radical from the group consisting of 1,4-bicyclo[2.2.2]octylene, piperidine-1,4-diyl, naphthalene-2,6-diyl, decahydronaphthalene-2,6-diyl and 1,2,3,4-tetrahydronaphthalene-2,6-diyl,

where the radicals (a) and (b) can be substituted by one or two fluorines,

20 Z<sup>1</sup> is -CO-O-, -O-CO-, -CH<sub>2</sub>O-, -OCH<sub>2</sub>-, -CH<sub>2</sub>CH<sub>2</sub>-, -CH=CH-, -C≡C- or a single bond, furthermore also -(CH<sub>2</sub>)<sub>4</sub>- or -CH=CH-CH<sub>2</sub>CH<sub>2</sub>-, and

m is 1, 2 or 3.

- 25 2. Liquid-crystal display according to Claim 1, characterized in that the medium contains at least one compound of the formula II



30 in which

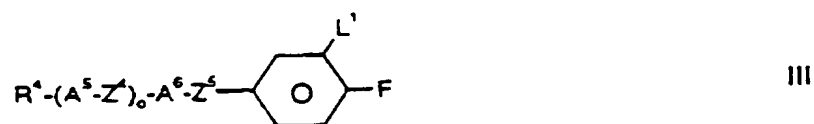
R<sup>3</sup> has the meaning given for R<sup>1</sup>,

A<sup>3</sup> and A<sup>4</sup> each have, independently of one another,  
the meaning given for A<sup>1</sup> and A<sup>2</sup>,

5 Z<sup>2</sup> and Z<sup>3</sup> each have, independently of one another,  
the meaning given for Z<sup>1</sup>, and

n is 0, 1 or 2.

10 3. Liquid-crystal display according to Claim 1 or 2,  
characterized in that the medium contains at least  
one compound of the formula III



in which

15 L<sup>1</sup> is H, F or Cl,

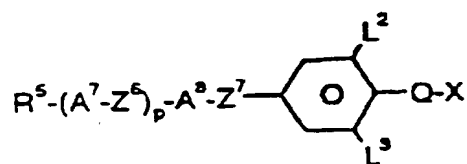
R<sup>4</sup> has the meaning given for R<sup>1</sup>,

20 A<sup>5</sup> and A<sup>6</sup> each have, independently of one another,  
the meaning given for A<sup>1</sup> and A<sup>2</sup>,

Z<sup>4</sup> and Z<sup>5</sup> each have, independently of one another,  
the meaning given for Z<sup>1</sup> and Z<sup>2</sup>, and

25 o is 0, 1 or 2.

30 4. Liquid-crystal display according to one of Claims 1  
to 3, characterized in that the medium contains at  
least one compound of the formula IV



IV

in which

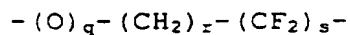
5  $R^5$  has the meaning given for  $R^1$ ,

$A^7$  and  $A^8$  each have, independently of one another, the meaning given for  $A^1$  and  $A^2$ ,

10  $Z^6$  and  $Z^7$  each have, independently of one another, the meaning given for  $Z^1$ ,

$L^2$  and  $L^3$  are each, independently of one another, H or F,

15  $Q$  is a polyfluoroalkylene radical of the formula



20 in which

$q$  is 0 or 1,

25  $r$  is 0 or an integer between 1 and 6,

$s$  is an integer between 1 and 6,

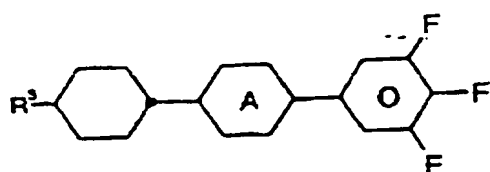
$X$  is H, F or Cl, and

30  $p$  is 0, 1 or 2.

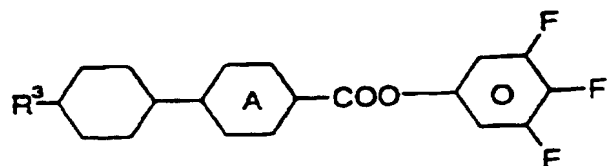
5. Liquid-crystal display according to one of Claims 1 to 4, characterized in that the medium contains not

more than 10% of liquid-crystalline compounds carrying cyano groups.

- 5        6.    Display according to one of Claims 1 to 5,  
characterized in that the pixels are addressed by  
an active matrix.
- 10       7.    Liquid-crystalline medium having positive  
dielectric anisotropy and containing a composition  
according to Claims 1 to 5.
- 15       8.    Liquid-crystalline medium according to Claim 7,  
characterized in that it contains
  - 20 to 60, preferably 15 to 40, % by weight of at  
least one compound of the formula I,
  - 10 to 60, preferably 20 to 50, % by weight of at  
least one compound of the formula II,
  - 20       - 0 to 20, preferably 5 to 15, % by weight of at  
least one compound of the formula III  
and
  - 25       - 0 to 40, preferably 20 to 30, % by weight of at  
least one compound of the formula IV.
- 30       9.    Liquid-crystalline medium according to Claim 5 or  
8, characterized in that it contains:
  - at least one compound selected from formulae  
IIa and IIb



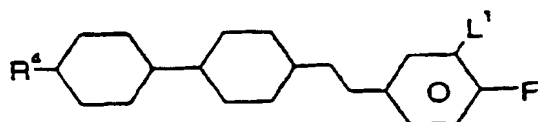
IIa



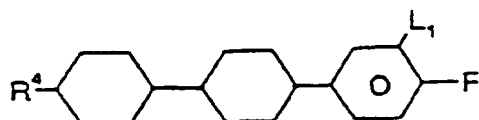
IIb

- at least one compound selected from formulae  
IIIa, IIIb, IIIc and IIId

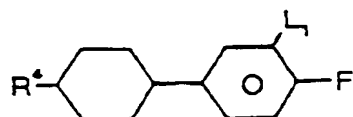
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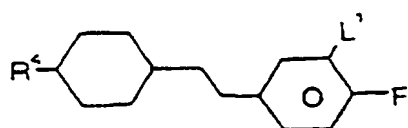
IIIa



IIIb

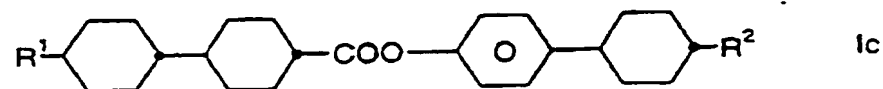
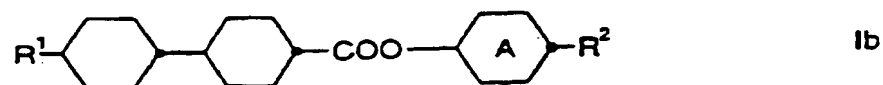


IIIc



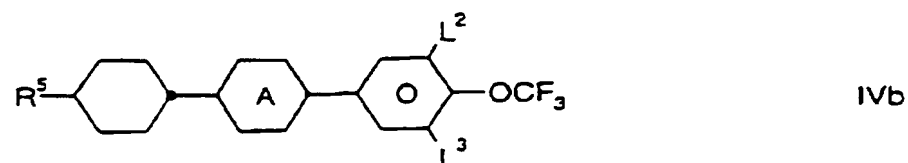
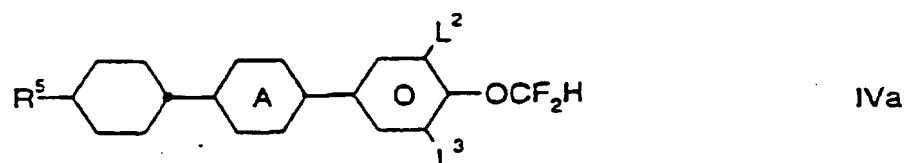
IIId

- at least one compound selected from formulae Ia,  
Ib and Ic

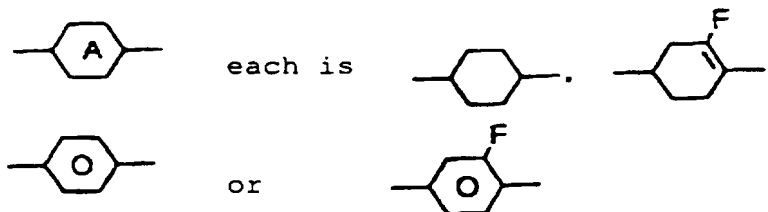


and, if desired,

- 5      - at least one compound selected from formulae IVa and IVb



in which



10

and R¹, R², R³, R⁴, R⁵, L¹, L² und L³ each have the meaning given.



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Application No: GB 9703021.7  
Claims searched: 1-9

Examiner: Stephen Quick  
Date of search: 13 May 1997

**Patents Act 1977**  
**Search Report under Section 17**

**Databases searched:**

UK Patent Office collections, including GB, EP, WO & US patent specifications, in:

UK CI (Ed.O): C4X (X12)

Int CI (Ed.6): C09K 19/00

Other:

**Documents considered to be relevant:**

Category	Identity of document and relevant passage	Relevant to claims
X	GB 2255417 A (MERCK PATENT), see whole document, especially example 1, page 23 (contains compounds of formula I)	7 at least
X	GB 2240987 A (MERCK PATENT), see whole document, especially example 3, pages 24 & 25 (contains compounds of formula I)	7 at least
X	GB 2234979 A (MERCK PATENT), see whole document, especially example 1, pages 14 & 15, media A & B (contain compounds of formula I)	7 at least
X	GB 2234507 A (MERCK PATENT), see whole document, especially example 9, pages 12 & 13, mixtures A & B (contain compounds of formula I)	7 at least
X	GB 2220658 A (VEB WERK FUR FERNSEHELEKTRONIK), see whole document, especially example 1, pages 8 & 9 (mixture of "GM + 1" with dielectric anisotropy of 1.695; contains compounds of formula I)	7 at least

X Document indicating lack of novelty or inventive step  
Y Document indicating lack of inventive step if combined with one or more other documents of same category.

& Member of the same patent family

A Document indicating technological background and/or state of the art.  
P Document published on or after the declared priority date but before the filing date of this invention.

E Patent document published on or after, but with priority date earlier than, the filing date of this application.



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Application No: GB 9703021.7  
Claims searched: 1-9

Examiner: Stephen Quick  
Date of search: 13 May 1997

Category	Identity of document and relevant passage	Relevant to claims
X	GB 2216523 A (MERCK PATENT), see whole document, especially example E, page 70 (contains compound of formula I at line 8)	7 at least
X	GB 2190675 A (VEB WERK FUR FERNSEHELEKTRONIK), see whole document, especially example I, page 8 (mixtures "GM+..." with positive dielectric anisotropy in lines 55-58, GM being basic mixture of lines 42-47; contains compounds of formula I)	7 at least
X	GB 2142647 A (SHARP KK), see whole document, especially claim 1 (contains compounds of formula I)	7 at least
X	GB 2122213 A (SHARP KK), see whole document, especially page 10 lines 13-14 and the positive dielectric anisotropies in table 1 (page 11) for the compositions in the table bridging pages 9 & 10 and the table on only page 10 (contain compounds of formula I)	7 at least
X	GB 2121406 A (SECRETARY OF STATE FOR DEFENCE), see whole document, especially page 14 (line 21) to page 15 (line 10) (ie mixtures 2 & 3; contain compounds of formula I)	7 at least

X	Document indicating lack of novelty or inventive step	A	Document indicating technological background and/or state of the art.
Y	Document indicating lack of inventive step if combined with one or more other documents of same category.	P	Document published on or after the declared priority date but before the filing date of this invention.
&	Member of the same patent family	E	Patent document published on or after, but with priority date earlier than, the filing date of this application.

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